PRODUCTION OF BIODIESEL FROM JATROPHA C. OIL USING KOH AND *LIPASE CANDIDA R.* IN AN AIRLIFT REACTOR

LIYANA BT AMER SHAH

DEGREE OF CHEMICAL ENGINEERING

(BIOTECHNOLOGY)

UNIVERSITI MALAYSIA PAHANG

UNIVERSITI MALAYSIA PAHANG

BORANG PENGESAHAN STATUS TESIS♦

JUDUL **PRODUCTION OF BIODIESEL FROM JATROPHA C. OIL USING KOH AND LIPASE C.R IN AIRLIFT REACTOR**

SESI PENGAJIAN : 2010/2011

Saya **LIYANA BT AMER SHAH**

(HURUF BESAR)

mengaku membenarkan tesis (PSM/~~Sarjana~~/~~Doktor Falsafah~~)* ini disimpan di Perpustakaan Universiti Malaysia Pahang dengan syarat-syarat kegunaan seperti berikut :

1. Tesis adalah hakmilik Universiti Malaysia Pahang
2. Perpustakaan Universiti Malaysia Pahang dibenarkan membuat salinan untuk tujuan pengajian sahaja.
3. Perpustakaan dibenarkan membuat salinan tesis ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (√)

☐	SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia seperti yang termaktub di dalam AKTA RAHSIA RASMI 1972)
☐	TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)
√	TIDAK TERHAD	

Disahkan oleh

(TANDATANGAN PENULIS) (TANDATANGAN PENYELIA)

Alamat Tetap **No. 59, Lrg 14,** **DR. IR. SAID NURDIN**

Tmn Assamaju 34000 Taiping, Nama Penyelia

Perak.

Tarikh : Tarikh:

CATATAN : * Potong yang tidak berkenaan.

** Jika tesis ini **SULIT** atau **TERHAD**, sila lampirkan surat daripada pihak berkuasa/organisasiberkenaan dengan menyatakan sekali sebab dan tempoh tesis ini perlu dikelaskan sebagai **SULIT** atau **TERHAD**.

♦ Tesis dimaksudkan sebagai tesis bagi Ijazah Doktor Falsafah dan Sarjana secara penyelidikan, atau disertasi bagi pengajian secara kerja kursus dan penyelidikan, atau Lapuran Projek Sarjana Muda (PSM).

"I hereby declare that I have read this thesis and in my opinion this thesis has fulfilled the qualities and requirements for the award of Degree of Bachelor of Chemical Engineering (Biotechnology)"

Signature	:	……
Name of Supervisor	:	DR IR SAID NURDIN
Date	:	……

PRODUCTION OF BIODIESEL FROM JATROPHA C. OIL USING KOH AND *LIPASE CANDIDA R.* IN AN AIRLIFT REACTOR

LIYANA BT AMER SHAH

A thesis submitted in fulfillment
of the requirements for the award of the Degree of
Bachelor of Chemical Engineering (Biotechnology)

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

DECEMBER 2010

I declare that this thesis entitled “Production of Biodiesel from *Jatropha C*. oil using KOH and *Lipase Candida R*. in an Airlift Reactor” is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.”

Signature :……

Name : LIYANA BT AMER SHAH

Date : ……

DEDICATION

Special dedication to all my friends,

My supervisor, Dr. Ir Said Nurdin, other lecturer,

FKKSA staffs and my beloved family.

For all your support, care, motivation and believe in me

ACKNOWLEDGEMENT

I am so thankful to Allah S.W.T for giving me patient and highly spirit completing this research project successfully done. With the mercifulness from Allah therefore I can produces a lot of information in this research project. To all my beloved freinds, I am so grateful to have you all in my life and giving me full of support in order to finish this project. A lots of thank to my parent for always standing behind me.

I am indebted to my supervisor, Dr Ir Said Nurdin, lecturer from the Faculty of Chemical and Natural Resources Engineering for her advice, insightful comments and support. Thank for your guide and without your guide this research will not be complete and well organized. My thanks also goes to Dr Reddy Prasad who which willing to share his knowledge on enzyme immobilization and to Prof. Badrulhisham for his kind heart to supplied the raw material for this research.

My sincere appreciation also extends to all my fellow colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Thank you for the time sacrificed to accompany me. And last but not least, I am grateful to all my family members.

ABSTRACT

Biodiesel was one of the alternatives fuels to fuel the vehicle since world petroleum depleted. Production of biodiesel from jatropha oil was attractive option compared to other edible oil such canola oil because it has no competed with food consumption issue. This research objective was to produce biodiesel from *Jatropha Curcas L.* using KOH and *Lipase Candida Rugosa* catalyst and to investigate the effect of reaction time, reaction temperature and methanol/oil ratio to biodiesel production. Two-stage transesterification method was used for KOH catalyst reaction while one-stage transesterification for biocatalyst reaction. Two-stage transesterfication method was the suitable method used to reduce high free fatty acid content in jatropha oil and increase the biodiesel yield. Without acid treatment in two-stage transesterification process, triglycerides tend to go through saponification reaction which produce soap rather than biodiesel. Methanol was used as solvent in both methods due to it short chain characteristic that can produce higher result than other type of alcohol. As a conclusion, biodiesel has been successfully produced from jatropha oil using KOH and Lipase Candida Rugosa enzyme and resulted 98% and 4% biodiesel yield respectively. The highest biodiesel production was obtained at 65°C at 1 hour of reaction and 6:1 methanol/oil ratio.

ABSTRAK

Biodiesel adalah salah satu alternatif sumber bahan bakar untuk kenderaan semenjak simpanan petroleum dunia berkurangan.Penghasilan biodiesel daripada minyak buah jarak merupakan pilihan yang menarik dibandingkan dengan minyak sayuran yang lain seperti minyak jagung kerana ia tidak mengalami persaingan dengan isu sumber makanan. Kajian ini dilakukan untuk menghasilkan biodiesel daripada minyak buah jarak jenis Curcas Lineus dengan mengunakan Kalium hyrodsida dan Lipase sebagai pemangkin. Dua peringkat transester proses digunalkan untuk tindak balas pemangkin Kalium hydrosida, manakala satu peringkat transester process untuk tindak balas pemangkin Lipase. Dua peringkat transester process penting untuk mengurangkan jumlah lemak tepu yang terdapat di dalam minyak buah jarak sekaligus meningkatkan penghasilan biodiesel. Tanpa penggunaan acid dalam transester proses dua peringkat, minyak buah jarak akan menghasilkan lebih banyak sabun berbanding biodiesel. Methanol digunakan sebagai pelarut di dalam kajian ini kerana rangkain karbonny yang pendek mampu meningkatkan penghasilan biodiesel berbanding jenis alcohol yang lain. Kesimpulannya, biodiesel telah berjaya dihasilkan daripada minyak buah jarak dengan mengunakan Kalium hydrosida dan Lipase sebagai pemangkin. Dengan menggunakan Kalium hydrosida 98% biodiesel telah terhasil manakala hanya 4% biodiesel terhasil hasil daripada penggunaan pemangkin Lipase. Jumlah biodiesel yang paling banyak dihasilkan pada suhu 65°C dalam masa 1 jam dan jumlah methanol kepada minyak buah jarak ialah 6:1.

TABLE OF CONTENT

LIST OF TABLE

LIST OF FIGURE

LIST OF APPENDIX

LIST OF SYMBOLS/ABBREVIATIONS

KOH	Potassium Hydroxide
°C	Degree celcius
FKKSA	Fakulti Kejuruteraan Kimia & Sumber Asli
µm	Micrometer
Wt%	Weight percent
GC	Gas Chromatography
MJ/m3	Mega joule per meter cube
Kg/m3	Kilogram per meter cube
ppm	Part per million
Et al	All other
LCR	*Lipase Candida Rugosa*
PBR	Packed Bed Reactor
MP	Methly Palmitate
MS	Methyl Stearate
H_2SO_4	Sulphuric Acid

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Biodiesel was one of the alternatives fuels to fuel the vehicle. Biodiesel was introduced to the world by Rudolph Diesel, a Germany Engineer on 1895which transform peanut oil to biodiesel (Demirbas, 2006) but, at that time the petroleum diesel based fuel still abundant thus make biodiesel fact hidden. After a few years later in 1970s, the world petroleum reserves depleted (Canakci, 2005) and the earth start fighting with the environmental problems. This make biodiesel was taken into consideration as one of the efficient alternatives fuel. Biodiesel was chosen as the alternative because it properties differ a bit from conventional diesel depended on the oil feedstock and alcohol used but very close to diesel fuel (Mittelbach et. al., 1992) Besides, biodiesel promising better advantages from petroleum based diesel in several aspects because it was renewable and environment friendly characteristics.

Currently, biodiesel is produced commercially in Europe and USA to reduce air pollution and the emission of greenhouse gas (Huofang, 2008). The global production of biodiesel had increased from 0.5 billion liters in a997 to 1.8 billion liters in 2003 (Demirbas, 2006).

Biodiesel was produced by the transesterification process of vegetable oil or animal fats with alcohol such methanol and ethanol (Knothe et. al., 2006). Biodiesel production process is sensitive to the quality of the feedstock requiring vegetable oil or animal fats with very low amount of FFA. The used of high quality feedstock contributed 80-88% of the overall production cost (Zhang., 2003). The most common feedstock for biodiesel is edible oil such as soybean, rapeseed, canola, sunflower, palm, coconut and even corn oil (Mondala, 2008).

Most of biodiesel was produced from edible oil but, the food competing issues with edible oil make non-edible oil was attractive. Jatropha oil is one of the non-edible oil. Jatropha was suitable raw material for biodiesel production since it can be used in cooking besides, have similar free fatty acid composition with other edible oil (Shah, 2004).

In biodiesel production there is several transesterification methods to converted vegetable oil into biodiesel. Since jatropha oil content high free fatty acids, two-step methanolysis method is more suitable which can reduced free fatty acid content before esterification process (Tiwari, 2007).

1.2 Problem Statement

In this study, jatropha curcas L. was selected as raw material for biodiesel production because the poison characteristic of jatropha oil which makes it have no competing issue compared to other edible oil. Besides, it was easy to grow in tropical and sub-tropical climate such Malaysia.

Most of biodiesel production used batch reactor with mechanical stirrer. But the suitable reactor for biodiesel production still has been searching in order to obtain the maximum product yield and reduce the biodiesel production cost.

The world demands for biodiesel keep increasing year by year this make researcher still searching the optimum parameter to obtain high yield, high quality and comparable biodiesel fuel with petroleum-based diesel.

Generally, the percentage of biodiesel yield from jatropha oil is still low due to the high free fatty acid content in jatropha oil.

1.3 Objectives

1. To produce biodiesel from Jatropha Curcas L. using KOH and Lipase Candida R.
2. To investigate the effect of temperature, reaction time, methanol-oil ratio on the biodiesel yield.

1.4 Scope of Research

To achieve the objective of this research, there are three scopes that have been identified:

1. Producing biodiesel from *Jatropha Curcas L.* using KOH and *Lipase Candida R.* in airlift reactor.
2. Evaluating the selected parameter for both KOH and Lipase catalyst (methanol/oil ratio, reaction temperature and reaction time).
3. Analyze the biodiesel produce using Gas Chromatography equipment.

CHAPTER 2

LITERATURE REVIEW

2.1 Biodiesel

Biodiesel refers to a vegetable oil- or animal fat-based diesel fuel consisting of long-chain alkyl (methyl, propyl or ethyl) esters. It produced from transesterification of vegetable oil or animal fats with alcohol. Biodiesel can be used alone or mixed with petroleum-based diesel as an alternative efficient fuel (Gerpen, 2005) and can be used in any diesel engine without modification (Progress and recent trends in biodiesel fuels., 2009). Biodiesel can be used as pure fuel or blended at any level with petroleum-based diesel for use by diesel engines. The most common biodiesel blends are B2 (2% biodiesel and 98% petroleum diesel), B5 (5% biodiesel and 95% petroleum diesel), and B20 (20% biodiesel and 80% petroleum diesel) (Balat, 2010).

Biodiesel have attractive characteristics compared to petroleum-based diesel because it is biodegradable, non-toxic, renewable and has reduced emission of CO, SO2, particulate matter, volatile organic compound and unburned hydrocarbons as compared to conventional diesel (Benjamin, 2008). But biodiesel also have several disadvantages such have high viscosity of vegetable oils as compared to petroleum-based diesel at 40°C leads to unfavorable pumping and spray characteristics (Sinha, 2008). As the result, it cause poor fuel atomization incomplete combustion, and carbon deposition on the injector and valve seats which give serious engine fouling (Kumar, 2010). Biodiesel offer safety benefits over diesel fuel because it is much less combustible, with a flash

point greater than 423 K compared to 350 K for petroleum-based diesel fuel (Balat, 2008).

2.1.1 Biodiesel Characteristics

Biodiesel is a light to dark yellow liquid. It is immiscible with water, has a high boiling point and low vapor pressure. Typical methyl ester has flash point around 150°C making it non-flammable. Selected properties of biodiesel compared to biodiesel fuels are shown in table below

Table: Specification of diesel and biodiesel fuel

Fuel property	Diesel	Biodiesel
Fuel standard	ASTM D975	ASTM PS 121
Fuel composition	C10-C21 HC	C12-C22 FAME
Lower heating value (MJ/m^3)	36.6×10^3	32.6×10^3
Kinematic Viscosity at 40°C (mm^2/s)	1.3-4.1	1.9-6.0
Specific gravity at 15.5°C	0.85	0.88
Density at 15°C (kg/m^3)	848	878
Water (ppm by wt)	161	0.05 % max
Carbon (wt %)	87	77
Hydrogen (wt %)	13	12
Oxygen (wt %)	0	11
Sulfur (wt %)	0.05 max	0.0-0.0024
Boiling Point (°C)	188-343	182-338
Flash Point (°C)	60-80	100-170
Cloud Point (°C)	-15 to 5	-3 to 12
Pour Point (°C)	-35 to -15	-15 to 10
Cetane number	40-55	48-65
Stoichiometric air/fuel ratio (wt/wt)	15	13.8

2.1.2 Biodiesel Standard

Biodiesel must meet American Society of Testing and Materials (ASTM) specifications. Standard specification for biodiesel fuel (B100) blend stock for distillate fuel is shown in Table 2.1. The purpose of this standard is to have biodiesel meet the performance requirement of engines without specifying the actual composition of the fuel. This will allow biodiesel to be produce from any feedstock as long as it meets the standard.

Table 1.1: ASTM standards of maximum allowed quantities in diesel and biodiesel (ASTM D6751-02).

Property	ASTM method	Limits	Units
Flash Point	D 93	130 min	°C
Water and sediment	D 2709	0.050 max	Vol %
Kinematic Viscosity, 40°C	D 445	1.9-6.0	Mm^2/s
Sulfated ash	D 874	0.020 max	Mass %
Sulfur	D 5453	0.05 max	Mass %
Copper strip corrosion	D 130	No. 3 max	-
Cetane number	D 613	47 min	-
Cloud point	D 2500	Report	°C
Carbon residue, 100% sample	D 4530	0.05 max	Mass %
Acid number	D 664	0.80 max	Mg KOH/g
Free glycerin	D 6584	0.02 max	Mass %
Total glycerin	D 6584	0.240 max	Mass %
Phosphorus content	D 4951	0.001 max	Mass %
Distillation temperature, 1 atm	D 1160	360 max	°C

Source: http://www.astm.org

2.2 Raw Material

The most common feedstock for biodiesel is edible oil such as soybean, rapeseed, canola, sunflower, palm, coconut and even corn oil (Mondala, 2008). The efforts to find alternative vegetable oil for biodiesel production with the aim to lowering the feedstock cost often suggested the utilization of waste cooking oil or non-edible vegetable oil which containing high amount of free fatty acid (FFA) (Nestor et. al., 2008).

Transesterification of rapeseed oil with candida rugosa lipase showed more than 99% of conversion (Wu, 1996). Biodiesel produced from soybean oil using rhizopus oryzae lipase resulted in 80-90% of conversion (Kaieda, 1999) and of sunflower oil with pseudomonas fluorescens lipase led to almost complete conversion of oil into biodiesel and glycerol (Iso, 2001).

2.2.1 Edible Oil

Biodiesel has been mainly produced from edible vegetable oils all over the world. More than 95% of global biodiesel production is made from edible vegetable oils (Demirbas, 2008).

2.2.1.1 Cottonseed Oil

Cottonseed oil was converted to biodiesel by alkali-catalyzed transesterification reaction at 0.75% catalyst concentration, 65°C reaction temperature, 6:1 methanol to oil molar ratio and 600 rpm of agitation speed. This optimum condition produced 96.9% of methyl ester.

2.2.2 Non-Edible Oil

The continuous and large-scale production of biodiesel from edible oils has recently been of great concern because they compete with food materials – the food versus fuel dispute. There are concerns that biodiesel feedstock may compete with food supply in the long-term (Refaat, 2009). Hence, use of non-edible vegetable oils when compared with edible oils is very significant in developing countries because of the tremendous demand for edible oils as food, and they are far too expensive to be used as fuel at present (Pramanik, 2003)

2.2.2.1 Cooking Waste Oil

Due to the high cost of the fresh vegetable oil, waste cooking oil attracted researcher to produce biodiesel from waste cooking oil because it is available with relatively cheap price (Nisworo, 2006: Zhang et. al., 2003). Although, the use of waste oils can lower the feedstock cost significantly, complicated procedures are needed to remove impurities, resulting in high operating costs (Al-Widyan, Bioresource Technology)

2.2.2.2 Jatropha Oil

Jatropha oil was extracted from jatropha plant. Jatropha Curcas Lineus was a natural wild plant, can grow without irrigation under a broad spectrum of rainfall (Foidl, 1996). Jatropha curcas is a genus comprising 70 species growing in tropical and sub-tropical country. It grows rapidly, takes approximately 2-3 years to reach maturity and it has lifespan in excess of 30 years. Jatropha contain high –seed yield and high oil content (Wood, 2005). The FFA composition in jatropha oil is similar to other edible oils but the presence of some anti-nutritional factors such as toxic phorbol ester makes this oil unsuitable for cooking purposes (Shah, 2004).

Jatropha pressed cake can be used as fertilizer and its organic waste products can be digested to produce biogas methane (Staubmann, 1997).

2.3 Catalyst

In conventional processes, biodiesel was produced by the transesterification of oils with alcohol in the presence of catalysts such as alkalis (KOH, NaOH) or their corresponding alkoxides (Jitputti, 2006).

Chemical catalysts including alkalis have been employed most widely since they give a high conversion of triglycerides to methyl esters in a short reaction time. However, chemical catalyst used in biodiesel production have several drawbacks including the difficulty of recycling glycerol, the need to eliminate the catalyst and salt leading to development of alternative pathways (Zhang, 2003). To overcome these problems an enzymatic process using extracellular or intracellular lipase catalyst has been developed (Kaieda, 1999). Biocatalyst is expensive than chemical catalyst but, reduced chemical consumption and waste water treatment cost in biodiesel production.

Biocatalyst is critically influenced by some parameters in biodiesel production such enzyme concentration, oil to alcohol ratio, water concentration, reaction temperature, reaction time and PH (Salis, 2005). Also, the enzymatic methylation do not present consistent conversions (Chen, 2010).